Table 1. Powder diffraction data

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Powder data

Diffractograms were recorded with a standard diffractometer; Cu Kα radiation (λ=1.5405 Å) was used. Powder diffraction data were given in Table 1.

Comparison with related structures

Yavapaiite structure, Graerer & Rosenzweig (1971). (Isomorphous compounds: 'anhydrous alums' of sodium NaAl(SO₄)₂, NaGa(SO₄)₂, NaV(SO₄)₂, NaFe(SO₄)₂, NaRh(SO₄)₂ and silver 'anhydrous alums' AgGa(SO₄)₂, AgCr(SO₄)₂, AgV(SO₄)₂, AgFe(SO₄)₂, AgRh(SO₄)₂.

Reference


Crystal data on 2AICl₃.3POCl₃. By PAUL H. COLLINS,* Department of Chemistry, The University, Southampton SO9 5NH, England

(Received 2 October 1974; accepted 6 November 1974)

Crystal data has been established for the compound 2AICl₃.3POCl₃. It is tetragonal, space group P4₂/m with cell dimensions a=b=19.39 (3) Å and c=13.05 (3) Å.

Origin of specimen

Aluminum trichloride was prepared by direct reaction between chlorine and aluminum powder and was purified by sublimation. A sample of this material was placed in a tube inside a dry box and excess redistilled phosphoryl chloride was added. The tube which was stoppered, became markedly warm. After the tube had stood for several hours, some colourless, acicular crystals had formed. These were sensitive to atmospheric moisture and so single crystals for X-ray examination were mounted in thin Pyrex capillaries inside the drybox.

Chemical analysis was not attempted as it would have been of doubtful value in distinguishing between the possible 1:2 and 2:3 addition compounds, the percentages of chlorine, aluminum and phosphorus all being rather similar for the two cases.

Crystal geometry and physical properties

Crystals were aligned about the needle axis (c). The cell dimensions, diffraction symmetry and systematic absences, l=2n+1 for 00l, were determined from room-temperature Weissenberg and precession photographs using Ni-filtered Cu Kα radiation (λ=1.5418 Å). They indicate that the space group is either P4₂ (No. 77) or P4₂/m (No. 84). Crystal data are given in Table 1. The experimental density was determined by flotation in bromoform/carbon tetrachloride mixtures.

Table 1. Crystal data

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<th>Formula</th>
<th>Al₂Cl₁₅O₃P₃</th>
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<td>c=13.05 (3) Å</td>
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<tr>
<td>D(measured)</td>
<td>1.94 ± 0.05 g cm⁻³</td>
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<td>D(calculated)</td>
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<tr>
<td>Systematic absences</td>
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Conclusions

The existence of several addition compounds has been postulated in the AlCl₃/POCl₃ system and there has been some measure of dispute as to their correct stoichiometries. The reported complexes are those with AlCl₃:POCl₃ ratios of 1:1 (Barabanova & Voitovich, 1964; Groeneveld & Zuur, 1957; Gutmann, 1952; Suvorov & Shubaev, 1971; Voitovich, Barabanova & Novitskaya, 1967), 1:2 (?) (Groeneveld & Zuur, 1957), 2:3 (Barabanova & Voitovich, 1964; Voitovich, Barabanova & Novitskaya, 1967) and 1:6, m.p. 43.5°C (Groeneveld & Zuur, 1957).

Melting-point determinations on the material under examination gave a value of 166 ± 1°C indicating that it was not the 1:1 adduct (a white solid, m.p. 186.5-188°C) but in
CRYSTAL DATA

fact either the 1:2 adduct or the 2:3 adduct (with literature
m.p.'s given as 164°C and 166°C respectively). These
stoichiometries were suggested on the basis of data from
thermal analyses and from thermal analysis/X-ray powder
studies respectively.

From experimental cell dimensions and the measured
density, Z can be calculated as very close to 8 for a 2:3
compound (2AlCl₃·3POCl₃; M.W. 726.7), or, 13.06 for a
1:2 compound (AlCl₃·2POCl₃; M.W. 440.0). Hence, this
appears to confirm the assignment of the compound as
2AlCl₃·3POCl₃.

No complete X-ray structure determinations have been
reported for adducts formed between AlCl₃ and POCl₃.
Difficulties are inherent because of the similar scattering
power of most atoms in the cell. Many crystal structures of
POCl₃ adducts have been published and in all cases so far
encountered phosphorus oxychloride acts as a donor
through the phosphoryl oxygen (e.g. Branden, 1962, 1963;

The author thanks Dr M. Webster for discussions and
the S.R.C. for a maintenance grant.

References
Inorg. Chem. 9, 1455–1456.
17, 353–361.
Pays-Bas. 76, 1005–1008.
Chem. 16, 166–170.
Voitovich, B. A., Barabanova, A. S. & Novitskaya, G. N.

Computer Programs


A computer program for molecular model construction. By M. W. Anker and A. Immirzi, Istituto di Chimica delle
Macromolecole, C.N.R., Via Alfonso Corti 12, 20133 Milano, Italy

(Received 13 May 1974; accepted 16 November 1974)

A computer program is described which assists in the construction of molecular models with spheres and rods
by using a two-circle drill.

This note describes a computer program designed to
assist in the construction of 'non-standard' molecular
models for compounds with irregular coordination geometries. In such cases ball and stick models may be constructed
from solid plastic spheres with holes drilled at the appro-
priate angles by means of the apparatus shown schematic-
ally in Fig. 1. The model is completed by connecting the
spheres with plastic or metal rods cut to the correct length.

The drilling device is available commercially* and con-
ists essentially of a horizontal drill and a two-circle
mounting for the plastic spheres. The horizontal \( \varphi \) arc
(Fig. 1) permits partial rotation of \( \pm 145^\circ \) about the z axis,
while the vertical \( \chi \) arc provides complete rotation about
the mounting axis.

To facilitate the use of this device, we have prepared a
computer program to calculate the required \( \chi \) and \( \varphi \) angle
settings and certain other constructional details directly
from the fractional cell coordinates and unit-cell param-
eters of the compound and the covalent radii of the ele-
ments present.

The program operates by considering each of the atoms
in the input list in turn as a central atom and searching for
every other atom within bonding distance (equivalent posi-
tions are not considered, but this feature would not be
difficult to incorporate). Two atoms are considered to be
bonded if their internuclear distance is less than the sum of
their atomic radii. After transformation of coordinates to
place the central atom at the origin, the \( \varphi \) and \( \chi \) angles are
calculated from the formulae:

\[
\begin{align*}
\varphi &= \arctan \left( \frac{z}{y} \right) \quad (\varphi = 0 - 360^\circ) \\
\chi &= \arccos \left( \frac{x}{d} \right) \quad (\chi = 0 - 180^\circ)
\end{align*}
\]

where \( x, y \) and \( z \) are Cartesian coordinates as defined in
Fig. 1, and \( d \) is the internuclear distance. If the \( \chi \) angle for
any bond exceeds the maximum permitted value (set by a
DATA statement to a conservative \( 140^\circ \)), the model is
rotated systematically until all the \( \chi \) angles fall within the
prescribed limit. It is unlikely that no suitable orientation
will be found, since this would imply that all nearest-
neighbour bond angles were less than about 60°.

In addition to the angle settings for the drill, the program

* For example from Charles Supper Co. Inc., 15 Tech
Circle, Natick, Massachusetts 01760, U.S.A. or from Ital-
structures, Riva del Garda, Italy.

Fig. 1. Schematic drawing of the drilling apparatus with def-
initions of the Cartesian axes and the \( \chi \) and \( \varphi \) angles.